A STUDY OF MICROSTRUCTURAL CHARACTERISTICS AND DIFFERENTIAL THERMAL ANALYSIS OF Ni-BASED SUPERALLOYS*

M.D. Aggarwal, R.B. Lal and S.A. Oyekenu
Department of Physics
Alabama A&M University
Normal, AL

and

Richard Parr and Stephen Gentz
Materials Laboratory, NASA/MSFC
Marshall Space Flight Center, AL

ABSTRACT

The objective of this work is to correlate the mechanical properties of the Ni-based superalloy MAR-M246(Hf) used in the space shuttle main engine, with its structural characteristics by systematic study of optical photomicrographs and differential thermal analysis. In this paper, we have developed a method of predicting the liquidus and solidus temperature of various nickel based superalloys viz., MAR-M247, Waspaloy, Udimet-4l, polycrystalline and single crystals of CMSX-2 and CMSX-3 and compared with the experimental DTA curves using Perkin-Elmer DTA 1700. The method of predicting these temperatures is based on the additive effect of the components dissolved in nickel. The results have been compared with the experimental values.

* Work supported under NASA Grant NAG8-076

A STUDY OF MICROSTRUCTURAL CHARACTERISTICS AND DIFFERENTIAL THERMAL ANALYSIS OF Ni-BASED SUPERALLOYS

Introduction

The purpose of this investigation is to study the microstructural characteristics of Ni-based superalloy MAR-M246(Hf) which is used in manufacturing the components of the space shuttle main engine. These superalloys are heat treated prior to their use. The heat treatment should be optimum to get the best performance. To find out the optimum heat treatment, the techniques of differential thermal analysis and optical photomicrographs are being utilized. Ni-based superalloys can be considered as "chemical stew" containing as many as 14 different elements.[1] Nickel serves as an ideal base for such alloys because of its high melting point, 1453°C, adequate corrosion resistance and ability to dissolve a number of other metallic elements which serve to strengthen it. The various elements go into the solid solution to provide different effects: Molybdenum, tantalum, and Tungsten go in to the alloy for strength; chromium, aluminum and tungsten add to oxidation resistance and γ' phase.

Differential thermal analysis (DTA) is a technique which continues to aid in defining the phase changes that occur in superalloys and is undoubtedly of considerable value to the metallurgist studying these complex systems. The effect of changes in major elements are easily determined. The DTA technique has proved to be a valuable technique for the superalloy metallurgist to study liquidus-solidus data, carbide precipitation reactions, γ' solvus temperatures and incipient melting temperatures.[2]

The width of the solidification range is an important casting parameter and has the controlling effect in the process of directional solidification. In a simple system, the equilibrium value of this quantity can be determined from the equilibrium diagram, but in more complicated systems like these, superalloys containing 12 to 14 elements, only approximate estimates can be obtained. In the present paper, we discuss a method of predicting the solidus and liquidus temperatures based on the additive effects of the components dissolved in nickel and the results are compared with the experimental results obtained from the DTA curves for various Ni-based superalloys such as MAR-M246(Hf), MAR-M247, Waspaloy, Udimet-41, polycrystalline and single crystals of CMSX-2 and CMSX-3.

Prediction of Solidification Range

For highly diluted systems as in Ni-based superalloys, Hayes and Chipman[3] gave a relation for the change of the melting point ΔT of the parent metal M after adding a small amount of component B:

$$\Delta T=(1-k_{0,B})\, N_{L,B}\, (T_M)^2/\Delta H_M \qquad (1)$$

where $k_{0,B}$ is the equilibrium distribution coefficient of component B in the parent metal M, ΔH_M is the heat of melting, T_M is the melting point of metal M (K), $N_{L,B}$ is the molar fraction of component B dissolved in the liquid phase of metal M, and R is the constant ($8.314\, J\, K^{-1}\, mol^{-1}$).

The equilibrium distribution coefficient $k_{0,B}$ is defined as T = constant by the relationship

$$k_{0,B} = N_{S,B} / N_{L,B} \tag{2}$$

where $N_{S,B}$ is the molar fraction of component B in the solid solution based on metal M.

For highly diluted solutions, the liquidus and solidus curves can be replaced by straight lines (Fig. 1) and the solidification range of the solution M-B, denoted by $I_B = T_{L,B} - T_{S,B}$, can be expressed by the relationship

$$I_B = (1-k_{0,B})\ \Delta T/k_{0,B} = (1-k_{0,B})^2\ T_M{}^2\ R\ N_{L,B}/k_{0,B}\ \Delta H_M . \tag{3}$$

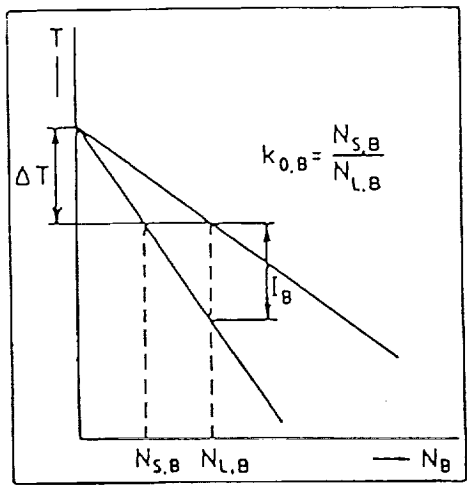

Fig. 1 Equilibrium diagram

If the metal is represented by nickel, Eq. (3) can be written in the form

$$I_B = 832.3\ (1-k_{0,B})^2\ c_B / k_{0,B}\ M_B \tag{4}$$

where c_B is the concentration of component B in weight percent, M_B is the molecular mass of component B, and I_B is in degree Celsius. If we assume the mutual independence of the effect of the components on the change of the melting point of parent metal M, the equilibrium solidification range I can be written as

$$I = \sum_B I_B . \tag{5}$$

Differential Thermal Analysis

In the differential thermal analysis (DTA) method, a reference sample and the sample under test are heated at a certain rate in a specific temperature range. The reference should exhibit no transformations within the temperature range of interest. The temperature of the sample and reference are monitored separately and the thermocouples measuring the temperature are connected in opposite polarity to measure the temperature differential ($\Delta T = T_{sample} - T_{reference}$). ΔT is amplified and then fed to Thermal Analysis Data Station (Perkin Elmer DTA 1700) which displays a plot of ΔT versus T for any sample. A schematic diagram of the DTA apparatus is shown in Fig. 2. Silver (melting point :

961 C) and nickel (melting point : 1455 C) are used to calibrate the instrument reasonably near our temperatures of interest. Sample holders/crucibles are 6 mm^3 cups made up of high density high purity alumina. Thermograms are stored in the computer's memory as well as displayed on the monitor which can be stored on floppy disks for later analysis. DTA curves of the various nickel based superalloys viz., MAR-M246(Hf), MAR-M247, Waspaloy, Udimet-41, polycrystalline and single crystals of CMSX-2 and CMSX-3 are compared with the calculated values using Eq. (5) neglecting the mutual interaction of the components.

Results and Discussion

Fig. 3 shows a typical DTA curve from which the values of solidus and liquidus can be determined. The onset for Ni-based superalloy UD-41 is 1293.8 C which represents the solidus and the minimum which represents the liquidus is at 1345.3 C. The solidification range is thus determined to be 52.5 C. Table 1 lists the values of c_B, $k_{0,B}$ and the solidification range of this alloy using Eq. (4). Table 2 lists the solidification range as obtained from DTA measurements and calculated using Eq. (5) for the other superalloys. The values for UD-41 are in better agreement than for the other superalloys. Further analysis of the data is in progress and will be reported elsewhere.

Acknowledgments

The authors wish to thank Mr. Richard J. Quigg, Vice-President of Cannon-Muskegon Corporation for providing us polycrystalline samples of CMSX-2 and CMSX-3. Thanks are due to Mr. Gregory Bell of Howmet Corporation for providing single crystal samples of CMSX-2 and CMSX-3. We also thank Mr. Sam O. Mancuso of Special Metals Corporation for providing us with samples of MAR-M247, UD-41 and Waspaloy. Thanks are also due to Dr. Bill Bhat, NASA/MSFC for useful discussion during the progress of this work.

References

1. Sims, C.T.; Stoloff, N.S. and Hagel, W.C., Eds. Superalloys (John Wiley, New York, 1987).

2. Wendlandt, W.W., Thermal Analysis (John Wiley, New York, 1986).

3. Hayes, A. and Chipman, J., Trans. AIME, 1939, 135, 85.

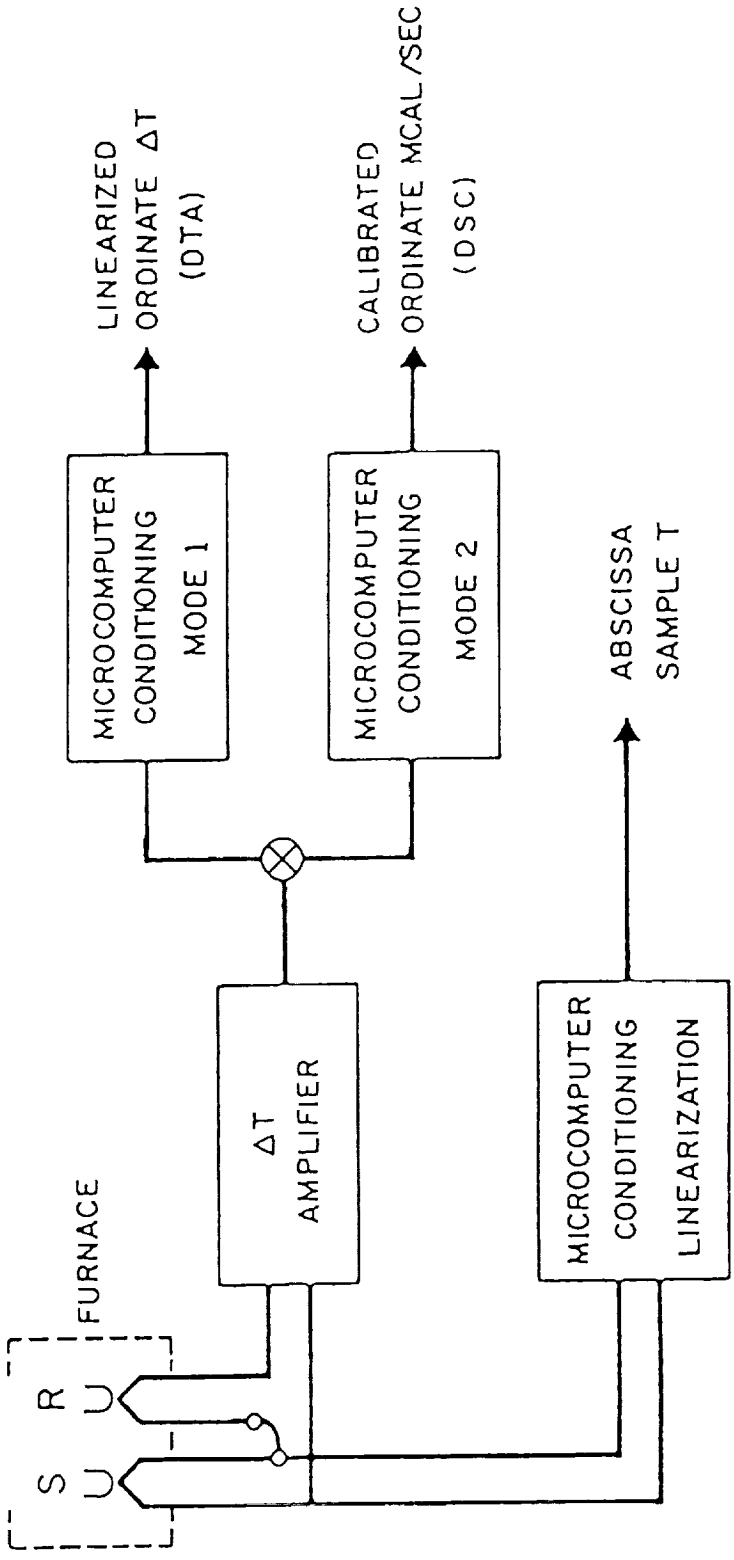
FURNACE
S
R
ΔT
AMPLIFIER
MICROCOMPUTER
CONDITIONING
LINEARIZATION
ABSCISSA
SAMPLE T
MICROCOMPUTER
CONDITIONING
MODE 1
MICROCOMPUTER
CONDITIONING
MODE 2
LINEARIZED
ORDINATE ΔT
(DTA)
CALIBRATED
ORDINATE MCAL/SEC
(DSC)

Figure 2.

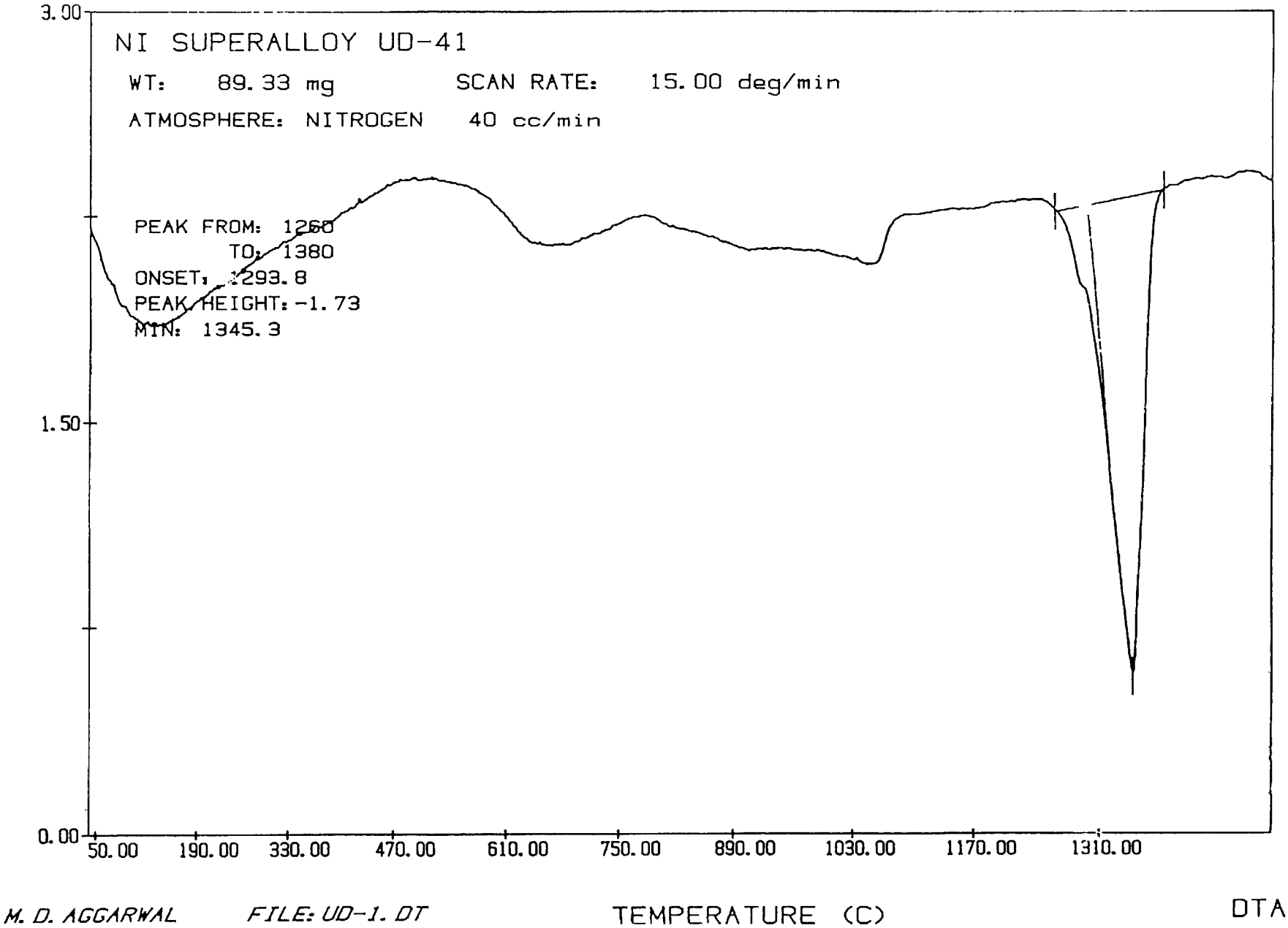
NI SUPERALLOY UD-41
WT: 89.33 mg
SCAN RATE: 15.00 deg/min
ATMOSPHERE: NITROGEN 40 cc/min
PEAK FROM: 1260
TO: 1380
ONSET: 1293.8
PEAK HEIGHT: -1.73
MIN: 1345.3
3.00
1.50
0.00
50.00
190.00
330.00
470.00
610.00
750.00
890.00
1030.00
1170.00
1310.00
M. D. AGGARWAL
FILE: UD-1. DT
TEMPERATURE (C)
DTA

Figure 3.

TABLE 1
Ni-based Superalloy UD-41

Element	C_B	$k_{0,b}$	M_b	Solidification range
C	0.08	0.22	12.01	15.33
Mn	0.10	0.72	54.94	0.16
Si	0.10	0.36	28.08	3.37
Cr	18.70	0.82	51.99	11.83
Ni	Bal	1.00	58.70	
Co	10.80	1.03	58.93	0.14
Mo	9.80	0.89	95.94	1.16
Ti	3.21	0.73	47.90	5.57
Al	1.59	0.87	26.98	0.95
Zr	0.07	0.09	91.22	6.20
P	0	0.01	30.97	8.85
Cu	0.01	0.82	63.55	0.01
Ta	0.01	0.74	180.95	0.00
			Total I	53.58

From DTA Curve

TS = 1293.8 C
TL = 1345.3 C

Diff. = 52.5 C

TABLE 2
Comparison of Experimental and Calculated Values of the Solidification Range.

Ni-based Superalloys	Solidification Range	
	Experimental (DTA)	Calculated (Eq.5)
UD-41	52.5 C	53.58 C
Waspaloy	40.3 C	52.49 C
CMSX-2 (Polycrystal.)	33.4 C	26.07 C
CMSX-3 (Polycrystal.)	39.8 C	26.315 C
CMSX-2 (single crystal)	32.9 C	25.97 C
CMSX-3 (single crystal)	36.2 C	25.95 C
MAR-M246(Hf)	44.4 C	56.31 C